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# Green extraction and dyeing of silk from Beta vulgaris peel dye with ecofriendly acid mordants

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**Abstract**

Natural dye from Beta vulgaris (Beetroot) peel was extracted ultrasonically and used to dye silk fabrics with and without ecofriendly acid mordants. The mordants used were citric acid, tartaric acid acetic acid and tannic acid. Colour fastness properties of dyed fabrics with different parameters (colour fastness to washing, colour fastness to acidic and basic perspiration, colour fastness to light, colour fastness to dry and wet rubbing, colour fastness to water and sea water, colour fastness to heat, dry cleaning and acid and alkali spotting) were assessed with the help of grey scale. Beetroot peel dye was found to have good fastness properties on silk fabrics. Dyed silk fabrics were also subjected to colour measurement values, Tristimulus (XYZ), LAB values and Munsell renotations (Hue, Lightness and Chroma). Hue values showed that Beetroot dye has blue colour.

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**Introduction**

The garden beet (Beta vulgaris, also known as the table beet or informally simple as beet is one of the many cultivated varieties of beets and arguably the most commonly encountered variety in North America and Britain. The art of making vegetable dyes is one of the oldest known to man and dates back, to the dawn of civilization. In India and Pakistan which are rich in natural resources these dyes were widely used for colouring of fabrics and different other materials (Thiyanarajan *et al.,* 2015; Samanta and Agarwal, 2009; Mahanata & Tiwari 2005). Although very earliest dyes were discovered accidently using berries and fruits. With the experimentation and gradual development the vegetable dyes has resulted into a very refined art expertise in vegetable dyes dates back to ancient time was suppressed. Some of the chemical dyes earlier found were associated with hazardous effects effecting human life creating skin diseases and lungs problems. (Adeel *et al.,* 2018; Khalid *et al.,* 2010; Cleinmen *et al.,* 1984).

Now there is growing demand of eco-friendly / non-toxics colorants specially for health sensitive applications such as coloration of food and dyeing of child textile / leather garments (Berhan *et al.,* 2017; Svinkumar *et al.,* 2009). So in recent years the inherent advantages of vegetables, fruits and flowers dyes has resulted in the revival and use of natural dyes. (Wang *et al.,* 2018; Kannadasan *et al.,* 2013 and Bechtold[a] *et al.,* 2003).

The common source of natural vegetable dyes are parts of plants such as leaves, flowers, fruits, seeds, barks and roots of dye yielding plants like, beetroot. Vegetable natural dyes do no cause any harm to human skin and no hazards are anticipated in their manufacturing rather some of the dyes acts as health cure. Chemical reaction is almost absent in the manufacture of vegetable dyes and there is no pollution problem and all these dyes are harmonized with nature (Nisa *et al.,* 2006; Waheed and Alam 2004; Michelle *et al.,* 2005). The application of natural vegetable dyes like beetroot, indigo, olive, logwood etc. in textile industry is the form of dyeing of yarns, dyeing of cloths (Silk, wool and cotton), dyeing of carpets and also in block printing (Ghias-u-deen *et al.,* 2015; Senad *et al.,* 2008; Pankage and Singh, 2005; Gulrajani and Gupta, 1992).

Similarly leather industry is also using vegetable dyes however the use is confined to cottage and small scale industries. At present none of the leather units are using vegetable dyes for coloration of their products (Khalid *et al.,* 2010; Velmurug *et al.,* 2010; Koralia and Dilliwar, 2009).

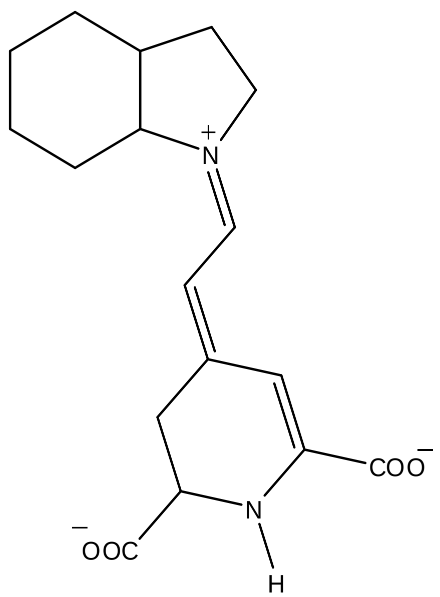
Most of the natural dyes generally require mordants for ensuring the reasonable fastness of the colour to sunlight and washing while some dyes are substantive and can be directly applied in textile fabrics cotton, wool, silk and leather without the need of any mordant.(Khalid *et al.,* 2008; Vankar *et al.,* 2007; Waheed and Alam, 2004; Deo and Desi, 1999).

Now the increasing interest to use natural dyes among public is due to the awareness of ecological and environmental problems related to the use of synthetic dyes. Natural dyes cuts down significantly on the amount of toxic effluents resulting from the synthetic dye processes. (Samanta *et al.,* 2009; Jain *et al.,* 2009).

Present work focus on the extraction of natural colourant from Beta vulgaris (Beetroot) peel, its application on silk fabrics and study of colour fastness and colour measuring properties with and without mordants. Mordants used were of acidic nature. (Amlepacil *et al.,* 2015; Khalid *et al.,* 2010; Waheed and Alam 2004). Previously a lot of work is done on the stability and bio-degradedly of beetroot. Extraction of natural dyes can be done with water or with different solvents (Sun[a], *et al.,* 2010; Fincan *et al.,* 2004; Valachovic *et al.,* 2001). Beta vulgarus is a herbaceous biennial or rarely perennial plant with leafy stems growing to 1-2 in tall. The leaves are heart-shaped, 5-20 cm long on plants. The flowers are produced in dense spikes. The fruit is a cluster of hard nutlets. All parts of the beet root contain oxalic acid (James and Duke, 1983).

The colour of the red/purple beetroot is due to a variety of betalain pigments. Some of the betalains in beetroot dye are betanin, isobetanin, probetanin and neobetanin which are collectively caused betacyanin. Other pigments contained in beetroot are betraxanthins (Azeredo *et al.,*., 2009; Kujala *et al.,* 2001).

Basic structure of betacyanins is:




Betanin a Betacyanin

Red beet root contains both red and yellow pigments of the class betanines. These are quaternary ammonium amino acids. Red colour is at its most intense and variable in beetroot from deep reddish-purple to bright vermilion but some varieties also have orange yellow and white roots. These colour difference are due to different levels of pigments (Betalains) that are characteristic of beetroot. (Kannan *et al.,* 2011; Azeredo *et al.,* 2009 Ddood *et al.,* 2001).

Beetroot red or betanin is used as a food colouring while beetroot juice is used for fabrics, leather and for hair colours (Qadariyah *et al.,* 2018; Kalporor *et al.,* 2008; Lio, 1981). Similarly betalains, betacyanins and betaxanthins which are water soluble pigments are used as food colors, these also have antioxiadant properties (Hadar *et al.,* 2018; Kristen *et al.,* 2006). Furthermore, these natural dyes are rich in nutrients which are important for the human health (Nisa *et al.,* 2006; Atamanova *et al.,* 2007; Jamnes and Duke, 1983).

Owing to favorable synthetic and application properties of synthetic dyes over natural dyes, at the end of 19th century natural dyes were almost completely replaced in the textile sector. Though synthetic dyes are available in wide range colors, which are higher reproducible and dyeing properties are improved at lower cost. But due to stringent environmental standards imposed by many countries in response to toxic and allergic reactions associated with synthetic dyes, these dyes are losing their place (Thomas Bechtold, 2006). As the world becoming more conscious towards ecology and environment, there is greater need today to revive the tradition of natural dye and dyeing techniques as an alternative of hazardous Synthetic dyes (K. Anitha, 2007). Natural dyes have now entered a new era where they are required to optimize their extraction and application procedures. Owing to vital role of natural plants as colorants for the textile, pharmaceutical and food industry, it is urgently needed to utilize zero cost dyes. Plants are the major sources of natural colorants and almost all their parts such as stem, leaves, fruits, seeds and pills are used for extracting natural color and they have antimicrobial, antifungal, insect repellent, deodorant, disinfectant and other medicinal values. In this study, the colorants were extracted from beta valgarus peels and applied with different mordants on silk. After dyeing, the dyed silk was subjected to different fastness tests.

## Materials and methods

Beet roots, Silk fabrics, Multiplefiber (DW), Grade 3 water, Detergent ECE (without optical brightener), sodium per borate, I-histidine monochloride monodhydrate, sodium dihyrogen orthophosphate, sodium carbonate, sodium hydroxide, citric acid, tartaric acid, acetic acid, and tannic acid. All chemicals used were of AR Grade.

*Collection of Plant Source*

Beetroots were purchased from local market of Lahore in Punjab province of Pakistan.

*Extraction of colourant*

Beetroots were peeled off about half centimeter depth, chopped and soaked in water for two hours so that material becomes soft.

Beakers with soaked dyestuff were then placed in ultrasonic bath and sonicated for 30 minutes at simmering temperature. After sonication, the contents of beaker were filtered through standard sieves of different mesh sizes.

The red colour solution was obtained which was dried and stored in an ambered colored bottle for further tests.(Qadariyah *et al.,* 2018; Kundal *et al.,*, 2016; Mishra *et al.,* 2012; Azeredo *et al.,* 2009).

*Preparation of Solutions*

2% and 4% strength solutions of beet roots peel dye were made to dye silk fabric. Similarly solutions of acid mordants (citric acid, tartaric acid, acetic acid and tannic acid) of 0.1M concentration were also made. (Khalid *et al.,* 2008; Pankag and Singh, 2005; Senad *et al.,* 2001).

*Substrate*

Silk fabric was obtained from silk industry near Lahore for dyeing.

*Scouring of Silk Fabric*

The scouring of silk fabric was done by washing it in a solution containing 0.5g/L sodium bicarbonate and 2g/L detergent at 50°C for 25 minutes. Keeping the material to liquor ratio at 1:40 scoured fabrics were thoroughly washed with tap water and dried at room.

*Dyeing without mordants*

For 2% and 4% shades, 10g of silk fabrics were taken for dyeing at the liquor ratio of 1: 25 for 45 minutes at 70°C by meta-mordanting technique in ultrasonic bath. After dyeing, the silk fabrics were removed and washed with hot and cold water.

*Dyeing with mordants*

0.1 M concentration of mordants (Citric acid, tartaric acid, acetic acid and tannic acid) were added during dyeing the silk fabric for fifteen minutes at 70-80°C by meta-mordanting technique in ultrasonic bath. After dyeing, the silk fabrics were removed and washed with hot and cold water (Haddar *et al.,* 2018; Baaka *et al.,* 2017).

*Colour Fastness to Washing*

Wash fastness of all the dyed fabrics was determined according to ISO 105 C06 method. Washing was done by preparing the soap solution containing 4g detergent and 1 g sodium per borate solution of distilled water. Then pH was adjusted to 10.5 +/- 0.1 by the addition of 1g of sodium carbonate. 10x 4cm of silk fabric pieces were attached to each Multifiber (DW) by sewing along with one of the shorter sides. The specimens were put into glasses of launder O-meter for 30 minutes at 60°C having liquor ratio 50:1.After 30 min samples were removed washed and dried at temperature not more than 60°C. The change in shade and stain were assessed with the help of grey scale. Results are shown in (Tables-I & I-A), (BS-1006).

*Colour fastness to perspiration*

Tests were carried out by dipping the fabrics into I-histidine monohydrochloride monohydrate solution according to ISO 105 E04 method. 4 x 10 cm of dyed silk fabric pieces were attached to each Multifiber by sewing along with one of the shorter sides and dipped separately into alkaline and acidic solutions for 30 minutes having liquor ration 50: 1. Then the silk specimens were placed in the Perspirometer kits and the desired pressure was applied. These kits were placed in the vacuum oven for 4 hours and then the kits were removed from the oven, dried at 60°C by hanging in air. Change in the colour and stain of each specimen was assessed with grey scale as shown in (Tables-2 & 2-A).

*Rubbing Fastness*

Rubbing fastness (dry and wet) test was carried out according to ISO 105 X 12 standard test procedure. Dry rubbing on silk fabrics was carried out with the help of Crock meter under a pressure of 9 N in to and fro movements on standard rubbing cloth. Both wrap and weft readings were noted. Change in shade was noted with the help of grey scale, values are shown in Table-4).

*Wet Rubbing*

Wet rubbing on silk fabrics was done under the same conditions of Crock meter as in the dry rubbing except the standard rubbing cloth was soaked into

100% deionized water. Change in shade was assessed with the help of grey scale, as shown in Table-4-A).

*Light Fastness*

Light fastness was carried out according to ISO 105 standard procedure BO2; on Weather O meter by Atlas.Xenon arc lamp was used which is an artificial light source representative of natural day light D65. Fabrics of measurement 7cmx12cm of silk fabrics were exposed to Xenon arc lamp for 24h, at standard testing conditions using blue wool as standard reference fabric. The treated silk fabrics were evaluated for colourfastness to light by comparing with grey scales.

*Colour fastness to Dry cleaning*

Colorfastness to dry cleaning was done with the help of solvent Perchloroethylene according to method ISO 105 DO1. Undyed cotton twill bags of 10cmx10cm measurements were stitched around three sides, Perchloroethylene solvent and agitated for 30 minutes at 30C.

After this the bags were removed from the container. The samples were squeezed to remove surplus solvent and dried in the air by hanging them at a temperature of 60 to 50°C.

Assessment of change in colour of samples and change in colour of solvent was carried out with the help of grey scale and silk fabrics of 4cmx10cm measurements were placed in separate bags along with 12 non-corrodable stainless steel disks and the fourth side of the bag was sewed. Then the bags were placed in the separate containers of Washtec containing 200ml of Perchloroethylene solvent.

*Colour fastness to Water and Seawater*

Clourfastness to water and sea water was evaluated in the same manner as for the colourfastness to perspiration. ISO 105 EOI and EO2 were used for water and sea water colourfastness respectively. For water, fabrics were dipped in deionized water.

While for colourfastness to seawater fabrics alongwith Multifibers were dipped in NaCl solution (30gm/L) for 30 minutes. For both water and sea water the above three treated composite fabrics were put in Perspirometer Kit. These kits were placed in the oven for 4h at 37±2°C. Then the specimens were dried at temperature not more than 60°C. Change in shade and in stain were noted with the help of grey scale.

*Colour fastness to spotting acids and alkali*

Spots of acetic acid 300g/l, Sulphuric acid 50g/l, Tartaric acid 100g/l and $Na_2CO_3$ 100g/l of water were put on the specimens and change in shade was assessed with ISO-105 AO2 grey scale.

*Colour fastness to Dry heat*

Dry hot pressing was done according to ISO 105 X 11. Specimens of silk fabrics were pressed at temperature 110+-2°C with hand iron and change in shade was assesses with grey scale (BS 2001). Results are shown in (Table 4).

*Colourfastness to Dry cleaning*

Colorfastness to dry cleaning was done with the help of solvent Perchloroethylene. 4 cm x10 cm of silk fabric was agitated in a bag of 12x12cm squared twilled cloth in perchloroethylene solvent. Change in shade of the silk fabric was assessed with the help of grey scale and change in colour of perchloroethylene solvent was assessed by putting the solvents in light cabinet.

*Colour measurements*

Readings of dyed silk fabrics were taken on Data colour measurement system. Tristimulus values (XYZ), ANLAB values and Munsell renotations (Hue, Lightness and Chroma) were determined (Khalid *et al.,* 2010). Results are shown in Table-7 & 7-A).

**Results and discussion**

Silk fabrics were dyed with Beta vulgaris (Beetroot) peel dye by ultrasonic method at 2% and 4% dyeing concentration solutions of using different acid mordants (citric acid, tartaric acid, acetic acid and tannic acid) (Konkachuichay *et al.,* 2002).

The results of fastness properties of beetroot peel dye on silk fabrics are given in following tables:

**Table 1.** Wash Fastness Properties of 2% Shade of Silk Fabric.

| Mordants | Diacetate | Cotton | Nylon | Polyester | Polyacrylic | Wool | Change in shade |
|---|---|---|---|---|---|---|---|
| Control (without mordants) | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 3 |
| Citric acid | 5 | 5 | 5 | 5 | 5 | 4-5 | 3 |
| Tartaric acid | 5 | 5 | 5 | 5 | 5 | 4-5 | 3-4 |
| Acetic acid | 5 | 4-5 | 5 | 5 | 5 | 4-5 | 3-4 |
| Tannic acid | 5 | 4-5 | 5 | 5 | 5 | 4-5 | 3 |

**Table 1A.** Wash Fastness Properties of 4% Shade of Silk Fabric.

| Mordants | Diacetate | Cotton | Nylon | Polyester | Polyacrylic | Wool | Change in shade |
|---|---|---|---|---|---|---|---|
| Control (without mordants) | 4-5 | 4-5 | 4-5 | 5 | 5 | 4-5 | 3-4 |
| Citric acid | 5 | 5 | 5 | 5 | 5 | 5 | 3 |
| Tartaric acid | 5 | 5 | 5 | 5 | 5 | 4-5 | 3-4 |
| Acetic acid | 5 | 4-5 | 4-5 | 5 | 5 | 4-5 | 2-3 |
| Tannic acid | 5 | 5 | 4-5 | 5 | 5 | 4-5 | 3-4 |

In control sample of 2% shade; diacetate, cotton, nylon, polyester and polyacrylic showed best (4-5) rating. For wool, the rating of change in stain was good (4) while change in shade of wool dyed without mordant was poor (3) change in stain of alum showed excellent (5) results for diacetate, nylon, polyester and polyacrylic whereas it showed best (4-5) results for cotton and wool while its change in shade was poor (3). Change in stain of citric acid gave excellent results (5) except wool which showed best results (4-5) and its change in shade was (3). Change in stain of tartaric acid also gave excellent results (5) except wool which gave best (4-5) rating. Change in shade of wool dyed with tartaric acid was satisfactory (3-4). Change in stain of acetic acid showed excellent results for diacetate, nylon, polyester and polyacrylic. For wool and cotton, it gave best results (4-5) while change in shade was (3-4) which was satisfactory. In control sample of 4% shade, the change in stain of polyester and polyacrylic was excellent (5) whereas diacetate, cotton, nylon and wool showed best (4-5) results, while its change in shade was satisfactory (3-4). For alum the change in stain of diacetate, cotton, polyester and polyacrylic gave excellent (5) results while nylon and wool gave best results (4-5) and its change in shade was satisfactory (3-4). Change in stain of citric acid showed excellent (5) results on all fabrics while its change in stain was (3). Tartaric acid showed excellent change in stain on diacetate, cotton, nylon, polyester and polyacrylic whereas its change in shade was satisfactory (3-4). Change in stain of acetic acid on diacetate, polyester and polyacrylic gave excellent (5) results while cotton, nylon and wool showed best (4-5) rating while its change in shade is 2-3.

**Table 2.** Acidic Perspiration of 2% Shade of Silk Fabrics.

| Mordants | Diacetate | Cotton | Nylon | Polyester | Polyacrylic | Wool | Change in shade |
|---|---|---|---|---|---|---|---|
| Control (without mordants) | 4 | 3-4 | 4-5 | 4-5 | 4-5 | 4 | 4 |
| Citric acid | 4 | 3-4 | 5 | 5 | 5 | 5 | 4 |
| Tartaric acid | 4 | 3 | 4-5 | 5 | 5 | 4-5 | 4 |
| Acetic acid | 4 | 3-4 | 5 | 4-5 | 4-5 | 4-5 | 3-4 |
| Tannic acid | 4-5 | 4 | 5 | 5 | 5 | 5 | 4-5 |

**Table 2A.** Basic Perspiration of 2% Shade of Silk Fabrics.

| Mordants | Diacetate | Cotton | Nylon | Polyester | Polyacrylic | Wool | Change in shade |
|---|---|---|---|---|---|---|---|
| Control (without mordants) | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 5 | 3 |
| Citric acid | 4-5 | 4 | 5 | 5 | 5 | 5 | 3 |
| Tartaric acid | 4-5 | 4 | 5 | 5 | 4-5 | 5 | 3 |
| Acetic acid | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 5 | 2-3 |
| Tannic acid | 4-5 | 4-4 | 5 | 5 | 5 | 5 | 4-5 |

In acidic perspiration, the change in stain of the wool sample dyed without mordant showed good (4-5) rating of nylon, polyester and polyacrylic whereas diacetate and wool gave (4) rating. Change in stain for cotton was satisfactory (3-4) while the change in shade of control sample was good (4). In case of alum the change in stain of nylon, polyester, polyacrylic and wool was excellent (5). For diacetate, the change in stain was (4-5) and for cotton it was (4) while its change in shade was best (4-5). Change in stain with citric acid showed excellent (5) results for nylon, polyester, polyacrylic and wool. For diacetate, its change in stain was (4) and for cotton it was 3 whereas the change in shade of wool dyed in the presence of citric acid as mordant was good (4). In case of tartaric acid, the change in stain of polyester and polyacrylic was excellent (5). For nylon and woolen the change in stain was best (4-5). For cotton its change in stain was (3) whereas diacetate showed (4) rating while its change in shade was good (4). In case of acetic acid the change in stain for polyester, wool and polyacrylic was best (4-5). Nylon showed excellent rating (5) whereas cotton gave (3-4) rating and diacetate give 4 while change in shade of silk fabric dyed with acetic acid as mordant was (3-4). In basic perspiration, the change in stain of the silk fabric sample dyed without mordant showed best (4-5) results for diacetate, cotton, nylon polyester and polyacrylic,. For woolen, change in stain was excellent (5) while its change in shade was (3). For alum change in stain of nylon, polyester, polyacrylic and wool was excellent (5). Change in stain of diacetate and cotton as best (4-5) while change in shade is also best. Change in stain of citric acid showed excellent (5) rating for nylon, polyester, polyacrylic and wool. For diacetate, change in stain was best (4-5) whereas cotton gave good (4) rating and its change in shade was (3). Change in stain for tartaric acid polyacrylic showed (5) results of nylon, polyester and wool whereas diacetate and polyacrylic showed (4-5) results. Change in stain for cotton was (4) while its change in shade was (3). For acetic acid the change in stain of diacetate, nylon, polyester and polyacrylic was best (4-5). For woolen change in stain was excellent (5) whereas cotton gave (4) rating and tis change in shade was (2-3).

**Table 3.** Acidic Perspiration of 4% Shade of Silk Fabrics.

| Mordants | Diacetate | Cotton | Nylon | Polyester | Polyacrylic | Wool | Change in shade |
|---|---|---|---|---|---|---|---|
| Control (without mordants) | 4 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4 |
| Citric acid | 4-5 | 4 | 5 | 5 | 5 | 5 | 4 |
| Tartaric acid | 5 | 4-5 | 5 | 5 | 5 | 5 | 4-5 |
| Acetic acid | 4 | 3-4 | 5 | 4-5 | 4-5 | 4-5 | 3-4 |
| Tannic acid | 4-5 | 4 | 5 | 4-5 | 5 | 5 | 4-5 |

**Table 3A.** Basic Perspiration of 4% Shade of Silk Fabrics.

| Mordants | Diacetate | Cotton | Nylon | Polyester | Polyacrylic | Wool | Change in shade |
|---|---|---|---|---|---|---|---|
| Control (without mordants) | 4-5 | 4-5 | 4-5 | 4-5 | 5 | 4-5 | 3-4 |
| Citric acid | 4 | 4-5 | 5 | 4-5 | 5 | 4-5 | 4 |
| Tartaric acid | 4-5 | 4-5 | 5 | 5 | 5 | 5 | 4-5 |
| Acetic acid | 4 | 3-4 | 5 | 5 | 4-5 | 4-5 | 3 |
| Tartaric acid | 4-5 | 4-5 | 5 | 5 | 5 | 4-5 | 4-5 |

In acidic perspiration the change in stain of the control sample of cotton, nylon, polyester, polyacrylic and wool showed best (4-5) results. Change in stain for diacetate was good (4) while its change in shade was also good (4). For alum the change in stain of nylon, polyester polyacrylic and wool gave excellent (5) results. Change in stain of diacetate and polyester was best (4-5) whereas cotton showed (4) rating and its change in shade was best (4-5). Change in stain for citric acid gave excellent (5) results rating for nylon, polyester, polyacrylic and wool. For diacetate, the change in stain gave (4-5) rating whereas cotton showed (4) while its change in shade was good (4). For tartaric acid the change in stain of diacetate, nylon, polyester, polyacrylic and wool have excellent (5) results. For cotton the change in stain showed best (4-5) rating and its change in stain of polyester, polyacrylic and wool showed best (4-5) results whereas nylon gave excellent (5) rating change in stain for cotton was satisfactory (3-4) while its change in shade was (3-4).

In basic perspiration, the change in stain of control sample was (4-5) for diacetate, cotton, nylon, polyester and wool whereas cotton showed excellent (5) results while the change in shade of control sample was satisfactory (3-4). For alum the change in stain of nylon, polyester, polyacrylic and wool was excellent (5) whereas diacetate and cotton showed best (4-5) results and its change in shade was also best (4-5). Change in stain for citric acid was best (4-5) for cotton, polyester and wool whereas nylon and polyacrylic gave excellent (5) results and its change in shade was (4). For tartaric acid the change in stain for nylon, polyester, polyacrylic and wool was excellent (5). Change in stain of diacetate and cotton was (4-5) while its change in shade was best (4-5). For acetic acid the change in stain of nylon and polyester was excellent (5) whereas polyacrylic and wool showed best (4-5) results, change in stain for cotton was (3-4) whereas for diacetate it was good (4). While change in shade of sample dyed with acetic acid was (3).

**Table 4.** Rubbing Fastness Properties of 2% Shade of Silk Fabrics.

| Mordants | Dry Rubbing | | Wet Rubbing | |
|---|---|---|---|---|
| | Warp | Weft | Warp | Weft |
| Control (Without mordant) | 4-5 | 4-5 | 4-5 | 4-5 |
| Citric Acid | 4-5 | 5 | 4-5 | 4 |
| Tartaric Acid | 4-5 | 4-5 | 4 | 4 |
| Acetic Acid | 4-5 | 4-5 | 4 | 4 |
| Tannic acid | 4-5 | 4-5 | 4 | 4 |

Change in shade in case of dry rubbing of silk fabrics dyed without mordant and with mordants (Citric acid, tartaric acid , acetic acid and tannic Acid) showed best (4-5). results change in shade in case of wet rubbing control gave best (4-5) rating showed good whereas citric acid, tartaric acid , acetic acid and Tannic Acid (4) results.

**Table 4A.** Rubbing Fastness Properties of 4% Shade of Silk Fabrics**.**

| Mordants | Dry Rubbing | | Wet Rubbing | |
|---|---|---|---|---|
| | Warp | Weft | Warp | Weft |
| Control (Without mordant) | 4-5 | 4-5 | 4 | 4-5 |
| Citric acid | 4-5 | 5 | 3-4 | 3-4 |
| Tartaric acid | 4 | 4-5 | 4 | 4-5 |
| Acetic Acid | 5 | 5 | 5 | 5 |
| Tannic Acid | 4-5 | 4-5 | 5 | 5 |

Change in shade for dry rubbing of silk fabric dyed without mordant and with mordants (Citric acid and tannic acid) showed best (4-5) results. Change in shade of tartaric acid was good (4) whereas for acetic acid it was excellent (5). Change in shade in case of wet rubbing for control was good (4). Change in shade for tannic acid and tartaric acid showed good (4) rating whereas citric acid gave satisfactory (3-4) results. Acetic acid showed best (4-5) change in shade.

**Table 5.** Light Fastness Properties of 2% Shade of Silk Fabric.

| Conc. of Dye | Mordants | Change in shade |
|---|---|---|
| 2% | Control (Without mordant) | 4 |
| | Citric Acid | 4 |
| | Tartaric Acid | 4 |
| | Acetic Acid | 4 |
| | Tannic Acid | 4-5 |
| 4% | Control (Without mordant) | 4 |
| | Citric Acid | 4 |
| | Tartaric Acid | 4 |
| | Acetic Acid | 4-5 |
| | Tannic acid | 4-5 |

For 2% dye concentration, change in shade of silk fabric dyed without mordant was good (4). Change in shade of silk fabric dyed with citric acid, tartaric acid and acetic acid as a mordant showed very good (4) rating whereas tannic acid gave best (4-5) results. For 4% dye concentration change in shade of wool dyed without mordant was good (4). Change in shade of wool dyed with the presence of mordant like alum, citric acid and tartaric acid also showed good (4) results whereas acetic acid gave best (4-5) rating. Results for colour fastness to Dry cleaning on silk Fabrics for both 2% and 4% were (5). For change in shade of Fabrics as well as for change in shade of solvents, no dye removal was found in Perchloroethylene solvent. Hence, results were Excellent. Colourfastness to water mostly showed rating from (4-5) to 5 of change in shade and stain of silk fabrics dyed with and without acid natural mordants with 2% and 4% dye concentrations. Colour fastness to sea water showed good to excellent results for dyed silk fabrics with 2% and 4% concentrations giving ratings (4), (4-5) and (5). Colour fastness to acid spotting gave (3-4) for sulphuric acid spotting on silk fabric dyed with acetic acid mordant for 2% shade which is satisfactory while others gave (4) and (4-5) rating which is good. Alkali spotting with Na2CO3 gave (3), (3-4) and (4) grey scale colour fastness to spotting test which were also satisfactory.

**Table 6.** Colour Fastness to Dry Cleaning of 2% and 4% Dyed Silk Fabrics with and without Mordants.

| Conc. of Dye | Mordants | Change in shade of Fabric | Change in shade of Solvent |
|---|---|---|---|
| 2% | Control(Without mordant) | 5 | 5 |
| | Citric Acid | 5 | 5 |
| | Tartaric Acid | 5 | 5 |
| | Acetic Acid | 5 | 5 |
| | Tannic Acid | 5 | 5 |
| 4% | Control(Without mordant) | 5 | 5 |
| | Citric Acid | 5 | 5 |
| | Tartaric Acid | 5 | 5 |
| | Acetic Acid | 5 | 5 |
| | Tannic Acid | 5 | 5 |

**Table 7.** Results of Colour Fastness to Water for 2% Shade.

| Mordants | Diacetate | Cotton | Nylon | Polyester | Polyacrylic | Wool | Change in Shade |
|---|---|---|---|---|---|---|---|
| Control Without mordant | 4-5 | 4 | 5 | 4-5 | 4-5 | 4-5 | 4 |
| Citric Acid | 5 | 4-5 | 5 | 5 | 5 | 4-5 | 4-5 |
| Tartaric Acid | 5 | 4-5 | 5 | 5 | 5 | 4-5 | 4 |
| Acetic Acid | 5 | 4-5 | 5 | 5 | 5 | 4 | 4 |
| Tannic Acid Acid | 4-5 | 4-5 | 5 | 5 | 5 | 4-5 | 4-5 |

**Table 7A.** Results of Colour Fastness to Water for 4% Shade.

| Mordants | Diacetate | Cotton | Nylon | Polyester | Polyacrylic | Wool | Change in Shade |
|---|---|---|---|---|---|---|---|
| Control Without mordant | 4-5 | 4 | 5 | 5 | 4 | 4-5 | 4 |
| Citric Acid | 4-5 | 4-5 | 4 | 5 | 4-5 | 4 | |
| Tartaric Acid | 5 | 4-5 | 4-5 | 5 | 5 | 4-5 | 4-5 |
| Acetic Acid | 5 | 4-5 | 4 | 5 | 4-5 | 4-5 | 4-5 |
| Tannic Acid | 5 | 4 | 4 | 5 | 5 | 5 | 4-5 |

**Table 8.** Colour Fastness to Seawater for 2% Shade of Silk Fabrics.

| Mordants | Diacetate | Cotton | Nylon | Polyester | Polyacrylic | Wool | Change in Shade |
|---|---|---|---|---|---|---|---|
| Control Without mordant | 4-5 | 4-5 | 5 | 4-5 | 4-5 | 4 | 4-5 |
| Citric Acid | 5 | 4-5 | 5 | 5 | 5 | 4-5 | 4-5 |
| Tartaric Acid | 5 | 4 | 5 | 5 | 5 | 4-5 | 4 |
| Acetic Acid | 5 | 4 | 5 | 5 | 5 | 4 | 4 |
| Tannic Acid | 4-5 | 4-5 | 5 | 5 | 5 | 4-5 | 4-5 |

**Table 8A.** Colour Fastness to Seawater for 4% Shade of Silk Fabrics.

| Mordants | Diacetate | Cotton | Nylon | Polyester | Polyacrylic | Wool | Change in Shade |
|---|---|---|---|---|---|---|---|
| Control Without mordant | 4-5 | 4 | 5 | 5 | 4 | 4-5 | 4 |
| Citric Acid | 4-5 | 4-5 | 4 | 5 | 4-5 | 4 | 4-5 |
| Tartaric Acid | 5 | 4-5 | 4-5 | 5 | 5 | 4-5 | 4-5 |
| Acetic Acid | 5 | 4-5 | 4 | 5 | 4-5 | 4-5 | 4-5 |
| Tannic Acid | 5 | 4 | 4 | 5 | 5 | 5 | 4-5 |

**Table 9.** Colour Fastness to Acid and Alkali spotting for 2% and 4% shade of Dyed Silk Fabrics.

| Conc. of Dye | Mordants | Acid Spotting | | Alkali Spotting |
|---|---|---|---|---|
| | | Acetic acid (300g/L) | Sulphuric Acid (150g/L) | Na2CO3 (100g/L) |
| 2% | Control Without mordant | 4 | 4 | 3 |
| | Citric Acid | 4 | 4 | 3 |
| | Tartaric Acid | 4-5 | 3-4 | 3 |
| | Acetic Acid | 4-5 | 4 | 4 |
| | Tannic Acid | 4-5 | 4 | 4 |
| 4% | Control Without mordant | 4-5 | 4 | 3 |
| | Citric Acid | 4-5 | 4 | 3-4 |
| | Tartaric Acid | 4 | 4 | 3 |
| | Acetic Acid | 4 | 4 | 4 |
| | Tannic Acid | 4-5 | 4 | 4 |

**Table 10.** Heat Fastness Properties of 2% & 4% shade of dyed silk fabrics.

| Conc. Of Dye | Mordants | Change in shade |
|---|---|---|
| 2% | Control (Without mordant) | 4 |
| | Citric Acid | 4 |
| | Tartaric Acid | 4 |
| | Acetic Acid | 4-5 |
| | Tannic Acid | 4-5 |
| 4% | Control (Without mordant) | 4-5 |
| | Citric Acid | 5 |
| | Tartaric Acid | 5 |
| | Acetic Acid | 4 |
| | Tannic Acid | 5 |

**Table 11.** Resuts of Colour Measurements of Silk Fabrics Dyed without Mordant for Beet Root Peel Dyed Fabrics.

| Sr. No | Conc. of Dye | Time of Dyeing | Temp. | Tristimulus | | | LAB values | | | Munsell Renotations | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | X | Y | Z | L | A | B | Hue | Lightness | Chroma |
| 1 | 2% | 45 min | 70°C | 49.15 | 49.30 | 46.08 | 70.21 | 2.07 | 10.23 | 43.0 | 70.2 | 9.0 |
| 2 | 4% | 45 min | 70°C | 46.06 | 46.46 | 45.10 | 68.16 | 1.33 | 8.48 | 49.5 | 68.1 | 7.1 |

For 2% dye concentration change in shade of silk fabric dyed without mordant was good (4). Change in shade of citric acid, tartaric acid and acetic acid were excellent (5). whereas. Tannic acid showed good (4) rating, for heat fastness properties.

*Colour measurement values of Beetroot Dye*

Results of colour measurements of silk samples dyes with and without mordants are given in the following tables. Tristimulus values (XYZ), ANLAB values (LAB) and Munsell renotation values (Hue, Lightness and Chroma) are noted. The colour coordinates XYZ, LAB and Munsell renotation values are noted for 2% and 4% dye solutions. Values of XYZ and LAB coordinated decreased with the increase or percentage shade as shown in table. Munsell values, Hue, Lightness and Chroma can also be seen in the above table. Hue (H) values increases while lightness and chromaticity decreases with the increase of percentage shade of Beetroot Peel dye. Chroma (C) increased with the increase of concentration shade of dye whereas lightness (L) decreased with the increase of concentration shade of dye.

**Table 11A.** Results of Colour Measurements of Silk Fabric Dyed with Beetroot Peel Dye with Mordants (Citric acid, Tartaric acid, Acetic acid and Tannic acid).

| Sr. No | Conc of Dye | Mord-ants | Mord-ant Conc. | Time in min | Tristimulus | | | LAB values | | | Munsell Renotations | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | X | Y | Z | L | A | B | Hue | Lightness | Chr-oma |
| 1 | 2% | Citric acid | 0.1 M | 45 | 41.39 | 40.01 | 36.18 | 63.25 | 6.10 | 10.36 | 63.0B | 63.2 | 10.6 |
| | 4% | Citric acid | 0.1 M | 45 | 35.58 | 35.09 | 18.15 | 59.23 | 3.54 | 23.29 | 23.8 B | 59.2 | 22.1 |
| 3 | 2% | Tartaric acid | 0.1 M | 45 | 37.59 | 36.59 | 24.69 | 60.48 | 5.06 | 18.14 | 14.5 B | 60.4 | 17.4 |
| | 4% | Tartaric acid | 0.1 M | 45 | 39.30 | 40.55 | 23.81 | 63.67 | 1.27 | 22.10 | 11.2 BG | 63.6 | 21.0 |
| 5 | 2% | Acetic acid | 0.1 M | 45 | 32.13 | 30.63 | 19.88 | 55.34 | 6.77 | 17.44 | 2..5 B | 55.3 | 17.2 |
| 6 | 4% | Acetic acid | 0.1 M | 45 | 30.67 | 29.35 | 15.68 | 54.17 | 6.24 | 20.16 | 4.6 B | 54.1 | 20.0 |
| 7 | 2% | Tannic acid | 0.1M | 45 | 40.92 | 40.15 | 30.83 | 63.36 | 4.38 | 15.50 | 57.7B | 63.3 | 14.7 |
| 8 | 4% | Tannic acid | 0.1M | 45 | 41.72 | 41.35 | 27.24 | 63.29 | 3.30 | 19.88 | 33.9 B | 64.2 | 18.7 |

The colour coordinates XYZ, LAB and Munsell renotation values are noted for 2% and 4% dye solutions dyed with mordants. Values of XYZ and LAB coordinates mostly decreased with the increase of percentage shade as shown in table. Munsell value Hue, Lightness and Chroma can also be seen in table. Hue (H) and Lightness (L) mostly decreased with the increase of concentration shade of dye while Chroma (C) increased with the increase of the concentration of dye. Most of the hue values are denoted by (B) which shows that beetroot peel dye contains blue colour except 4% shade of tartaric acid which show BG (Blue and green colour).(Waheed and Ashrarf, 2003; Waheed *et al.,* 1996; Waheed and Ashraf, 2003; Waheed *et al.,* 2004).

## Conclusion

Extraction and dyeing with *beta vulgaris* (Beetroot) peel natural dye was studied with ultrasonic greener technique with and without mordants on silk fabrics instead of old conventional exhaust method. The dye obtained was found to show rating of (3) good fastness properties with ecofriendly acid mordants (citric acid, tartaric acid acetic acid. and tannic acid) when applied by simultaneously mordanting technique. The colour measurement values of dyed fabrics showed that beetroot dye has blue colour.

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